

Los Alamos National Laboratory is operated by the University of California for the United States Department of Energy under contract W-7405-ENG-36

TITLE: Low Density Inorganic Foams Fabricated Using Microwaves

LA-UR--85-508

DE85 007708

AUTHOR(S): Thomas T. Meek, MST-6  
Rodger D. Blake, MST-6  
Thomas G. Gregory

SUBMITTED TO: American Ceramic Society Proceedings of Conference Held  
January 21-27, Cocoa Beach, FL on Composites

#### DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

**MASTER**

By acceptance of this article, the publisher recognizes that the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or to allow others to do so, for U.S. Government purposes.

The Los Alamos National Laboratory requests that the publisher identify this article as work performed under the auspices of the U.S. Department of Energy

 **Los Alamos** Los Alamos National Laboratory  
Los Alamos, New Mexico 87545

*[Handwritten signature]*

# LOW DENSITY INORGANIC FOAMS FABRICATED USING MICROWAVES

BY

T. T. MEEK, R. D. BLAKE, T. G. GREGORY

LOS ALAMOS NATIONAL LABORATORY

## ABSTRACT

The objective of our work was to determine if high temperature foams could be made using microwave heating; and if so, to investigate some of their properties. Several foams were made and their compressive strengths, tensile strengths and densities were determined. Foams were made of glass, metal-glass, glass-fiber, metal-glass-fiber, and fly ash. The microwave source used was a Litton model 1521 microwave oven which operated at 2.45 GHz and had an output of 700 watts.

## Introduction

The history of microwave heating of oxide materials dates back at least to the work of Von Hippel in the late 1940's. His interest was in how the loss characteristics of these materials varied as a function of frequency. Over the last 25 years little has been published in this area; however, work has been done at GMC,<sup>3-7</sup> and at the University of Edmonton in Edmonton, Alberta Canada by W. Tinga.

This paper addresses some preliminary work done at Los Alamos on the fabrication of low density structures using microwave heating. Several glass, glass-ceramic, and glass-metal-ceramic systems were investigated and will be discussed in this paper. Some mechanical properties will be reported on along with microstructural information.

## Preparation of Foams

Table I lists the different foams prepared and Table II shows the composition of some foams. It was decided to fabricate foams initially from glass microballoons and metal microballoons using a glass binder (in this case potassium silicate) which also acted as a microwave coupling agent and foaming agent. The glass microballoons were obtained from 3M Company and were a typical soda lime silicate glass with size distribution of from 30 microns to 130 microns. The metal microballoons were obtained from International Harvester Company and were composed of mainly nickel with some silicon and manganese. Their size ranged from around 400 microns to 600 microns. Also used to make foams was fly ash microballoons, a by produce of coal fired electrical generating facilities. Table VII shows the exact composition of the microballoons used. Several foams initially were made by mixing various microballoons with potassium silicate and then heating in a microwave oven. Densities of these foams are listed in Table VI, the F series foams are the first generation foams and the T series foams are the second generation foams that have been fabricated using microwave heating.

From Table II it is seen that many materials were used as microwave coupling agents. A coupling agent is a material that at room temperature will absorb microwave energy at 2.45 GHz (usually materials with OH, CO, NO, or NH bonds). This material will heat at 2.45 GHz and raise the surrounding temperature of the ceramic or glass thus increasing their loss tangent and increasing their microwave coupling efficiency. Along with the requirement that the coupling agent couple well to 2.45 GHz microwave radiation, the coupling agent also had to form a strong bond with the foam material in order to improve the foam mechanical properties.

Typical fabrication procedure was to mix by weight microballoons as shown in Table I with a suitable coupling agent as shown in Table III. The mixture was then placed into a reaction cavity inside a model 1521 Litton microwave oven (shown in Fig. 1) and heated for between five minutes and 30 minutes in air.

## Foam Microstructures

Figures II-IV show SEM photomicrographs of several of the foams discussed above. In all cases the coupling agent glass used formed excellent bonds with the microballoons used. Of interest is the wetting of the glass binder material and the metal microballoons. Excellent adherence is seen between the glass binder and the metal microballoons yet relatively low temperatures were measured (less than 600°C for ten minutes). Also of interest were the foams in which were used SiC fibers. Mechanical strength data indicates no beneficial effect of the SiC fibers on foam integrity. This is probably because processing temperatures were not high enough to form strong bonds between the microballoons, glass binder and the SiC fibers. Also fiber geometry was not optimized to improve packing density. Table 3 lists the coupling agent binder glasses used. The higher temperature binder glasses generally resulted in improved mechanical properties.

## Mechanical Properties

The sample configurations shown in Fig. V and Fig. VI were used to obtain compressive strength and tensile strength data on the foams listed in Table I.

As seen in Table IV the strongest foams in tension were T-5, T-8, and T-13 and in compression were T-12, T-11, T-10, and T-7 as shown in Table V. All of these foams had as a major phase fly ash except for T-5 and T-7 which were composed of glass and metal microballoons and either a low temperature glass binder with potassium silicate or just potassium silicate used as a coupling agent and a binder itself. A variable that differed from samples T-5 and T-7 and the fly ash samples was exposure time to the microwave radiation. The fly ash foams were heated for fifteen minutes while the non fly ash foams were heated only six minutes. Probably higher temperatures were reached in the fly ash foams; however, this is not known for certain because no good method has been found for measuring temperature in a microwave environment. If appreciably higher temperatures were achieved in the fly ash foam then phases could have been formed which resulted in superior mechanical properties.

The lowest density foams were made using either fly ash ( $0.09 \text{ g/cm}^3$ ) or metal microballoons with potassium silicate, also  $0.09 \text{ g/cm}^3$ .

## Conclusions

The work described in this paper is preliminary in nature and much further work needs to be done to better relate observed mechanical properties to processing parameters. Much more detailed microstructural analysis needs to be done to correlate structure with foam composition, and foam mechanical properties. It has been demonstrated, however that inorganic low density foams can be fabricated using microwave energy, and mechanical properties obtained to date indicate that these foams can be machined and used perhaps for structural components in space.

Further work to improve foam tensile and compressive strength is in progress. By varying the microwave processing time and foam binder composition and using proper geometry fibers, better foam mechanical properties may result.

#### Acknowledgements

The authors wish to thank Mr. Richard Bare for his excellent work in preparing foam samples for tensile and compressive strength analysis.

## References

1. A. R. VON HIPPEL, "Dielectric Materials and Application" (John Wiley and Sons, New York, 1954).
2. A. R. VON HIPPEL, "Dielectrics and Waves" (John Wiley and Sons, Inc., 1954).
3. W. R. TINGA, "Interactions of Microwaves with Materials" Proceedings of IMPI, Short Course for Users of Microwave Power Nov. (1970).
4. W. R. TINGA, S. O. NELSON, "Dielectric Properties of Materials for Microwave Processing - Tabulated", Journal of Microwave Power, Vol. 8 (73), (1973).
5. W. R. TINGA and W. A. G. VOSS, "Microwave Power Engineering", Academic Press, New York, 2, (1968).
6. W. R. TINGA AND E. M. EDWARDS, "Dielectric Measurement Using Swept Frequency Techniques", Journal of Microwave Power, 3 (1968) 144-175.
7. W. R. TINGA, "Multiphase Dielectric Theory - Applied to Cellulose Mixtures", Thesis, Dept. of Electrical Engineering, University of Edmonton, Edmonton, Alberta, (1969) 62-65.

- Figure I - Microwave reaction cavity used to prepare the foams.
- Figure II - Shown is a 200X view of a broken glass bond between two metal microballoons (Trade name Solacel).
- Figure III - Shown is a 2000X view of glass bonds between glass microballoons.
- Figure IV - Shown is a 2000X view of glass bonds between glass microballoons and a metal microballoon.
- Figure V - Shown is a machined foam sample ready for obtaining tensile strength data.
- Figure VI - Shown is a machined foam sample ready for obtaining compressive strength data.

TABLE I  
FOAMS FABRICATED USING MICROWAVE HEATING

FLY ASH + C

GMB + C

GMB + SiC FIBERS + C

GMB + MB + C

GMB + MB + FIBERS + C

MB + C

MB + FIBERS + C

GMB = glass microballoons

MB = metal microballoons

C = coupling agent



TABLE II  
COMPOSITION OF SELECTED FOAMS

F-1	Potassium Silicate - 22.8 g 3M Glass Bubbles - 9.7 g
F-2	Potassium Silicate - 22.8 g 3M Glass Bubbles - 8.5 g SiC Fibers - 0.9 g
F-3	Potassium Silicate - 22.8 g 3M Glass Bubbles - 7.3 g SiC Fibers - 0.9 g Metal Beads - 0.5 g
F-4	Potassium Silicate - 27.8 g 3M Glass Bubbles - 7.3 g Metal Beads - 0.5g
F-5	Potassium Silicate - 22.8 g 3M Glass Beads - 4.9 g Metal Beads - 3.1 g
F-6	Potassium Silicate (enough potassium silicate was used to bind the metal microballoons together) Fly Ash - 57.0 g Hydrated $\text{KSiO}_4$ - 61.2 g

TABLE III  
COUPLING AGENTS USED

GLYCEROL

NITRATES

POTASSIUM SILICATE

SODIUM SILICATE

1613 GLASS

01 0338 GLASS

01 1756C GLASS

TABLE IV  
TENSILE STRENGTH OF FOAMS

SAMPLE	TENSILE STRENGTH	
	MPA	(psi)
T-1-1	1.03	(149)
-2	1.24	(180)
-3	1.01	(146)
T-2-1	1.17	(170)
-2	1.21	(176)
-3	1.10	(159)
T-3-1	0.93	(135)
-2	0.78	(113)
-3	0.70	(101)
T-4-1	0.99	(144)
-2	0.68	(98)
-3	0.24	(34.5)
T-5-1	1.21	(176)
-2	2.76	(400)
-3	3.24	(470)
T-6-1	0.74	(108)
-2	1.13	(164)
T-7-1	1.56	(226)
T-8-1	2.34	(340)
-2	1.76	(255)
-3	0.62	(90)
T-9-1	0.93	(135)
-2	0.39	(56)
-3	0.81	(118)
T-10-1	0.54	(54)
-2	0.81	(118)
-3	1.01	(146)
T-11-2	1.35	(196)
T-12-1	1.09	(158)
T-13-1	0.66	(96)
-2	1.05	(153)
-3	2.41	(350)
T-14-1	1.35	(196)
-2	0.34	(50)
-3	1.35	(196)
T-15-1	0.74	(107)
-2	0.50	(73)

TABLE V  
COMPRESSIVE STRENGTH OF FOAMS

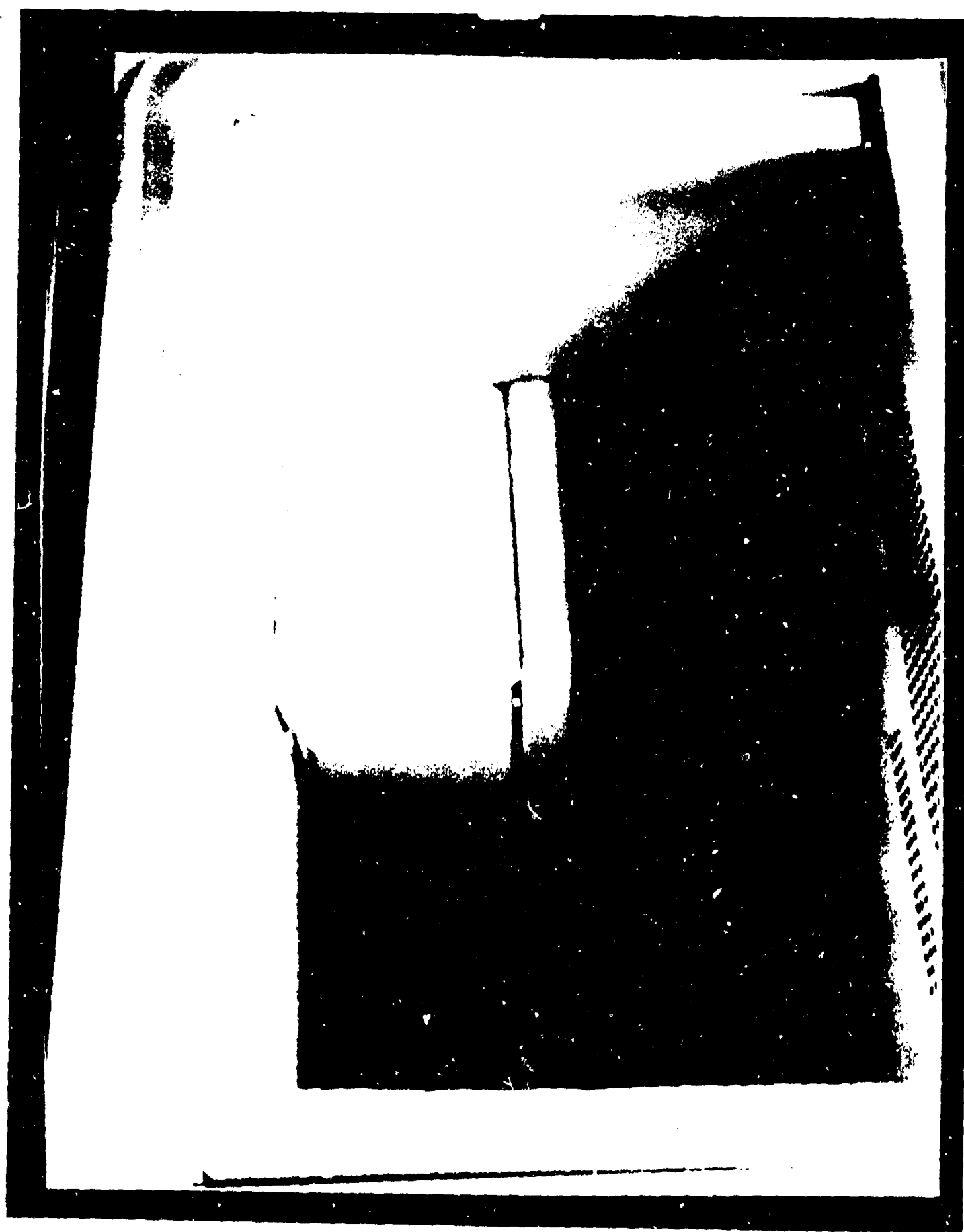
SAMPLE	COMPRESSIVE STRENGTH	
	MPA	(psi)
T-4-4	2.79	(405)
-5	3.17	(460)
T-5-4	3.79	(550)
-5	3.79	(550)
T-6-4	3.17	(460)
-5	2.96	(430)
T-7-2	3.96	(575)
-3	4.38	(635)
T-8-4	3.10	(450)
T-9-4	2.41	(350)
-5	2.96	(430)
-6	3.07	(445)
T-10-1	4.76	(690)
T-11-3	4.89	(710)
-4	2.52	(365)
T-12-2	4.07	(590)
-3	5.38	(780)
T-13-4	3.34	(485)
-5	3.41	(495)
-6	2.90	(420)
T-14-4	2.86	(415)
-5	2.55	(370)
-6	2.86	(415)
T-15-3	3.79	(550)
-4	3.86	(560)

TABLE VI  
FOAM DENSITIES

SAMPLE #	DENSITY g/cc
F1	0.14
F2	0.32
F3	0.30
F4	0.28
F5	0.24
T1	0.30
T2	0.34
T3	0.33
T4	0.19
T5	0.27
T6	0.09
T7	0.32
T8	0.11
T9	0.09
T10	0.13
T11	0.14
T12	0.12
T13	0.16
T14	0.41
T15	0.40

TABLE VII  
Microballoon Composition

Microballoon	Material	Weight %
Glass	$\text{Na}_2\text{O}$	5.6
	$\text{SiO}_2$	85.3
	$\text{CaO}$	9.1
Solacel	Ni	74
	Mn	24
	Si	2
Fly ash	$\text{SiO}_2$	57.1
	$\text{Al}_2\text{O}_3$	31.2
	$\text{K}_2\text{O}$	5.4
	$\text{FeO}$	5.2
	$\text{TiO}_2$	1.1





200X





**2,000X**



**2,000X**

